

# Pilot-plant distillation of meadowfoam fatty acids<sup>☆</sup>

Steven C. Cermak \*, Terry A. Isbell

*New Crops Research, National Center for Agricultural Utilization Research, Agriculture Research Service, USDA,  
1815 N. University St., Peoria, IL 61604, USA*

Received 16 January 2001; accepted 1 August 2001

## Abstract

Crude meadowfoam fatty acids, which are mainly long chain monoenes,  $\Delta$ -5 C20, were separated using a Myers 15 pilot plant centrifugal molecular distillation unit to give a distillate that was light in color (Gardner Color = 1). One of the problems with crude meadowfoam fatty acids is the color (Gardner Color = 18). The optimal distillation conditions were explored by varying the rotor temperature, degas temperature, rotor preheat, and flow rate onto the rotor. As the conditions were varied, the distillate was monitored for color, fatty acid composition, and mass split between distillate and residue. At very high rotor temperatures, the color of the distillate and the fatty acid composition deteriorated, but the same split was observed as with a low rotor temperature. All the variable conditions played a vital role in conducting a successful distillation. In most cases, direct correlations existed between temperatures and amounts of different fatty acids. The optimum conditions were determined through the course of numerous trials and used to distill a large quantity (95 l of meadowfoam fatty acid). © 2002 Elsevier Science B.V. All rights reserved.

**Keywords:** Meadowfoam fatty acids; Pilot plant; Distillation; Molecular still; Centrifugal molecular distillation; Decolorization

## 1. Introduction

Interest in meadowfoam has developed because of the unique long-chain unsaturated fatty acids present in the oil. Meadowfoam oil main compo-

nents were 5-eicosenoic acid (64%), 5,13-docosadienoic acid (19%), 5-docosenoic acid (3%), and 13-docosenoic acid (10%). Meadowfoam oil has many interesting physical properties such as an unusually high oxidative stability index compared with other vegetable oils (Isbell et al., 1999) and is mainly composed of monoenoic fatty acid, which helps increase its oxidative stability.

Previous studies of meadowfoam oil and the fatty acids hydrolyzed from the oil have resulted in several novel compounds. Estolides (Isbell et al., 1994) and lactones (Isbell and Plattner, 1997)

<sup>☆</sup> Names are necessary to report factually on available data; however, the USDA neither guarantees nor warrants the standard of the product, and the use of the name by USDA implies no approval of the product to the exclusion of others that may also be suitable.

\* Corresponding author. Tel.: +1-309-681-6233; fax: +1-309-681-6524.

E-mail address: cermaksc@ncaur.usda.gov (S.C. Cermak).

are two such compounds from meadowfoam. Meadowfoam estolides have cosmetic applications as they improve conditioning, shine, and comb-out compared with existing conditioners (Isbell et al., 2000). One of the problems with meadowfoam estolides is their color. Ideal cosmetic ingredients should be colorless (Gardner = 0). Crude meadowfoam fatty acids are very dark (Gardner = 15+), which leads to very dark estolides (Gardner = 18+). If crude meadowfoam fatty acids could be purified to a low Gardner color at a low cost, this would lead to a lower colored estolide and eliminate post-distillation decolorization (Frykman and Isbell, 1999). Improvement in meadowfoam estolide would help the commercialization of this new oil seed and give US farmers a valuable rotation crop.

Meadowfoam fatty acids can be successfully separated at relatively low temperatures to produce nearly colorless fatty acids. Many compounds are sensitive to heat such as high vacuum oils (Rees, 1980), vegetable oils, pharmaceuticals, and cosmetics (Batistella and Maciel, 1996), so conventional distillations cannot be used with these materials. Molecular or short-path distillation, which has been known for a long time (Biehler et al., 1949), uses high vacuum to achieve distillation of thermally unstable materials. This approach is usually the most economically feasible method of purification. Centrifugal and falling films are two basic types of molecular distillation units, which use a short exposure of the distilled liquid on the evaporating surface. The high temperature exposure time in these stills is on the order of a few seconds to tens of seconds as the liquid is spread evenly in the form of a film (Micov et al., 1997). These types of distillation units have been used successfully to demonstrate and compare the distillation of many different compounds, such as carotenoids from palm oil (Batistella and Maciel, 1998).

The objective of this study is to investigate the general conditions, heating element temperatures, flow, and vacuum necessary for pilot-plant centrifugal distillation of crude meadowfoam fatty acids. Fatty acid profile, Gardner colors, and power requirements are examined to determine the best set of operating conditions.

## 2. Materials and methods

### 2.1. Material

Meadowfoam fatty acids were supplied by the Fanning Corp. (Chicago, IL). The fatty acid methyl ester (FAME) standard mixtures were obtained from Alltech Associates, Inc. (Deerfield, IL) and NuCheck (Elysian, MN). Concentrated sulfuric acid was obtained from T.J. Baker Chemical Co. (Clifton, NJ). Methanol and hexanes were obtained from Fisher Scientific Co. (Fairlawn, NJ). Ethanol was obtained from Aaper (Shelbyville, KY).

### 2.2. Equipment and procedures

#### 2.2.1. Gas chromatography

Gas chromatography (GC) was performed with a Hewlett–Packard 5890 Series II gas chromatograph (Palo Alto, CA), equipped with a flame-ionization detector and an autosampler/injector. Analyses were conducted on a SP 2380 30 m × 0.25 mm i.d. column (Supelco, Bellefonte, PA). Saturated C<sub>8</sub>–C<sub>30</sub> FAMES provided standards for calculating equivalent chain length (ECL) values, which were used to make fatty acid and lactone assignments.

SP 2380 analysis: column flow 3.3 ml/min with helium head pressure of 103.42 kPa; split ratio 22:1; programmed ramp 150–185 °C at 7 °C/min, 185–265 °C at 15 °C/min injector and detector temperatures set at 250 °C. Retention times for eluted peaks with ECL values in parentheses: methyl-5-eicosenate 6.51 min (20.37), methyl-5-docosenate 8.30 min (22.29), methyl-13-docosenate 8.47 min (22.51), and methyl-5,13-docosadienate 8.72 min (22.85).

#### 2.2.2. Gardner color

Gardner color was measured on a Lovibond 3-Field Comparator from Tintometer Ltd. (Salisbury, England) using AOCS method Td 1a-64 (Firestone, 1994). Gardner color of both the residue and distillate materials was measured throughout the distillation. The + and – notation was employed to designate samples that did not match one particular color or, in the case of

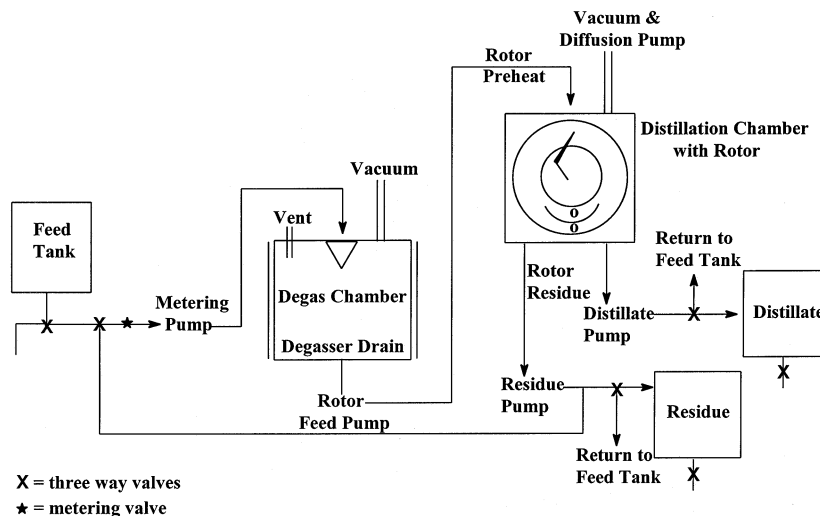


Fig. 1. Operational flow diagram for the molecular distillation unit.

the residue, an 18 + represented a color darker than the upper limit of 18.

### 2.2.3. GC analysis hydroxy fatty acids

Analytical samples for GC were prepared by heating a 10 mg sample of distillate or residue in 1 ml of 1 M  $\text{H}_2\text{SO}_4/\text{MeOH}$  to reflux in a sealed vial, placed in a heating block for 30 min. The solution was transferred to a separatory funnel with water (1 ml) and washed with hexanes ( $2 \times 2$  ml), dried over sodium sulfate, gravity filtered, placed in a GC vial, sealed, and injected into the GC.

### 2.2.4. Pilot-plant molecular distillation

The Myers Pilot-15 was used. It is a continuous centrifugal 38.1 cm, (15 in.), molecular still that contains all of the components needed for distilling raw feedstock (Fig. 1). Raw feedstock is delivered with a metering valve and a gear pump. It first enters a degasser unit maintained at pressures between 0.72 and 0.91 Pa and then to the heated evaporator cone or distillation chamber, where the molecular distillation takes place. The distillation and residue are continuously removed by transfer pumps. The material is passed to and from each station through stainless-steel transfer lines, which can be traced with heating tape. This unit was designed for use in extended or large scale distillations.

The major component of the 38.1 cm centrifugal molecular still is a concave, heated, evaporator cone, which rotates within the distillation chamber and is maintained at pressures as low as  $< 0.009$  Pa (Fig. 2). Two vacuum, mechanical-vane pumps and a high-vacuum diffusion pump supply the degas and distillation chambers. Degassed and/or stripped feedstock is metered by the feed pump into the center of the spinning (1700 rpm) evaporator cone and is spread rapidly and evenly outward in a thin film over the entire surface by centrifugal forces. As the film spreads and is heated, part of the feedstock reaches a temperature at which it vaporizes and leaves the rotor surface to condense on the cooler surface of

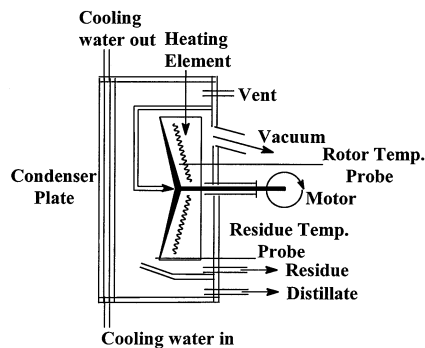


Fig. 2. Side view of distillation chamber.

the condenser plate. With the help of centrifugal force the unvaporized feedstock, the residue, is spun into a gutter, where it is removed from the still by a constant-speed transfer pump. The condensed vapor, the distillate, flows by gravity into a removal pipe located at the bottom of the distillation chamber and then pumped from the still by another constant speed transfer pump.

The operational distillation unit has two temperature sensors in the distillation chamber (Fig. 2). The first sensor, which monitors the temperature of the rotor, is located by the electric heating elements just beneath the 38.1 cm rotor. This first temperature actually reflects the temperature of the heating element, not the temperature of the rotor. The actual temperature of the rotor can be changed by varying the flow of feed material without affecting the element temperature. The second and most important temperature sensor reflects the temperature of the residue. This sensor is located in the gutter where the residue is collected during distillation. The residue sensor provides the closest temperature of the rotor, since it is a measurement of the temperature of the material that was just in contact with the rotor.

All the transfer lines and pumps are wrapped with heating tape. The heating tape and electrical elements are controlled with digital controllers either by percent power or actual measured temperature. Each region of the transfer line and pump has internal temperature probes that measure the temperature of the material as it passes the sensors. The temperatures are displayed on the control panel and are recorded during each distillation.

#### *2.2.5. Pilot-plant molecular distillation—startup*

System vents are closed and mechanical pumps are started to evacuate the system (Figs. 1 and 2). The cold traps are filled with liquid nitrogen to condense any volatiles. The system is allowed to pump down until the distillation chamber reaches about 0.91 Pa, at which point the diffusion pump is activated. As the diffusion pump comes on-line, a slight increase in the pressure of the distillation chamber occurs due to the degassing of the diffusion pump oil. Once

the distillation chamber reaches about 0.09 Pa, the metering valve is opened and the metering gear, rotor feed, and residue pumps are engaged. The degas chamber, rotor, transfer lines and pumps are slowly warmed up to distillation temperatures. As the temperatures are brought up to the set temperature, the system is placed in recycle mode. Since no material is being removed by distillation, the material is returned to a point prior to the metering valve via the residue pump. Recycling the initial material provides a pre-heating effect. This aids in the heating of the unit by eliminating the introduction of cold material. Once distillation begins, the distillate transfer pump is activated and the first sample out of the distillate transfer tube is discarded. After distillation begins and all set points are achieved, the recycle condition is canceled and residue is expelled from the residue transfer tube and again the first sample is discarded. Finally, the flow rate is adjusted to obtain a desired residue and distillate split.

#### *2.2.6. Pilot-plant molecular distillation—standard conditions*

The standard pressure conditions for the distillation of meadowfoam fatty acids from crude material are: chamber pressure  $\leq 0.009$  Pa, degasser chamber pressure  $\leq 0.91$  Pa.

The standard preset temperature conditions for this distillation of meadowfoam fatty acids from crude meadowfoam fatty acids are: rotor 450 °C, rotor preheat 130 °C, degasser chamber 150 °C, degasser feed 74 °C, degasser drain 110 °C, distillate drain 40 °C, residue line 80 °C, and rotor feed pump 105 °C. These set conditions directly affect the rotor residue temperature of 155 °C. The exact temperature settings are not always observed on the digital displays, the temperatures are a function of flow and heat transfer, so they have the tendency to fluctuate (Tables 1, 3 and 5).

The standard flow rate for the distillation of meadowfoam fatty acids from crude meadowfoam fatty acids is 150 g/min. An increase in the flow rate will lower many of the tempered regions, and thus, decrease the distillate to residue split ratio.

Table 1  
Distillation conditions-varying rotor temperature

Set point temperature (°C)	Observed rotor temperature (°C)	Rotor residue (°C)	Rotor preheat (°C)	Degasser drain (°C)	Rotor feed pump (°C)	Distillate to residue ratio	Flow (g/min)
275	275	129	113	101	85	0.2	111
325	325	144	113	101	85	0.7	98
375	374	145	114	103	86	1.9	102
400	400	146	121	108	95	4.0	109
425	424	167	114	104	86	16.8	99
475	475	244	114	105	87	42.3	90

Table 2  
Main components-fatty acid profile-varying rotor temperature

Temperature (°C) <sup>a</sup>	20:1 Δ5-d	20:1 Δ5-r	22:1 Δ5-d	22:1 Δ5-r	22:1 Δ13-d	22:1 Δ13-r	22:2 Δ5,13-d	22:2 Δ5,13-r	Gardner Color <sup>b</sup>
S.M. <sup>c</sup>	59.9 <sup>c</sup>		4.3 <sup>c</sup>	–	17.3 <sup>c</sup>	–	11.9 <sup>c</sup>	–	15 <sup>c</sup>
275	72.0	56.9	2.0	4.9	5.8	13.4	10.1	18.9	3
325	69.2	54.1	2.6	5.6	7.3	15.3	11.9	20.2	1
375	69.6	40.1	2.8	7.4	7.9	20.2	12.9	26.1	1
400	63.2	38.2	3.4	8.9	9.4	23.7	14.6	27.1	1
425	59.7	35.0	4.3	7.1	12.0	19.4	17.4	23.8	1
475	59.2	35.2	4.4	6.6	12.2	18.0	17.5	21.9	4

<sup>a</sup> Desired rotor temperature set point.

<sup>b</sup> Distillate.

<sup>c</sup> Starting crude meadowfoam fatty acids.

Fatty acid profile of distillate (-d) and residue (-r).

Table 3  
Meadowfoam distillation-varying rotor preheat temperature

Set point temperature (°C)	Observed rotor preheat (°C)	Rotor residue (°C)	Rotor feed pump (°C)	Distillate to residue ratio	Flow (g/min)
100	99	149	93	2.0	108
110	110	148	93	2.5	110
115	115	148	94	2.9	109
130	129	148	95	4.0	108
140	140	146	96	5.2	108
150	149	148	98	6.9	107

Rotor temperature constant at 400 °C.

### 2.2.7. Pilot-plant molecular distillation—shutdown and clean-up

When material in the feed tank falls below 1.9 l, the rotor, transfer lines, degas chamber heat, and the diffusion pump are turned off. Residual heat in the system will continue to successfully distill the remaining material. As the last of the material is drained from the feed tank, the metering valve is closed and, once the material has passed through the chamber, the degas transfer pump is turned off. Finally, the rotor motor and transfer pumps are turned off. All valves and gates are closed to maintain a closed system. Once the rotor has cooled to room temperature, which is the slowest component to reach equilibrium, the system is then vented to the atmosphere. When the distillation chamber is vented before the rotor has cooled, the residue material on the rotor will thermally decompose on the rotor to make clean-up difficult.

The clean-up of meadowfoam fatty acids requires that the transfer lines and pump be heated to about 40 °C to help melt any solids. At this temperature the remaining material in the lines melts and is easily removed with ethanol. About 3.8 l of ethanol is added to the feed tank and is pumped throughout the system. Hexanes are then added to the feed tank followed by another round of ethanol as described. The degas and distillation chambers are completely disassembled and cleaned with soap and water. The rotor sometimes exhibits a thick residue stain resulting from harsh distillation conditions of dirty feedstock. With the use of a mild abrasive, the stainless steel rotor returns to its original shine. The degas and distil-

lation chambers are reassembled and a final ethanol wash is pumped throughout the system. The system is vented, all valves, and gates are closed and the mechanical pumps are brought on-line. The system is allowed to pump down until all the ethanol has been collected in the cold traps and drained. At this point, the system vacuum gauges should show normal operating conditions, indicating that the system has no leaks. If the system does not reach these conditions, the two chambers may have to be readjusted until a leak free system is obtained.

### 2.2.8. Pilot-plant molecular distillation—sampling schedule

All data (Tables 1–6) were collected by setting the instrument to the desired conditions then allowing the instrument to reach equilibrium. The amount of time required for equilibrium depended on the set of conditions being modified and if they were being increased or decreased. Individual data points were collected over a given time, usually 1–2 h, and then averaged.

## 3. Results and discussion

Crude mixtures of meadowfoam fatty acids were separated using a Myers 15 pilot plant molecular centrifugal distillation unit. A series of conditions were examined to identify the optimum operating conditions including: rotor temperature, degas temperature, rotor preheat, and flow to the rotor. The main heating source for the distillation unit is the rotor element, which is located beneath

Table 4

Main components-fatty acid profile-varying rotor preheat temperature

Temperature (°C) <sup>a</sup>	20:1 Δ5-d	20:1 Δ5-r	22:1 Δ5-d	22:1 Δ5-r	22:1 Δ13-d	22:1 Δ13-r	22:2 Δ5,13-d	22:2 Δ5,13-r	Gardner Color <sup>b</sup>
100	65.5	43.7	3.0	7.6	8.4	20.5	13.4	26.0	1
110	65.0	42.8	3.1	8.1	8.7	21.4	13.7	24.6	1
115	65.0	41.8	3.3	8.7	9.0	23.0	14.2	25.8	1
130	62.4	37.3	3.4	9.3	9.2	24.5	14.3	27.3	1
140	62.7	30.6	3.5	10.3	9.8	26.5	15.1	25.1	1
150	59.5	43.2	4.0	9.0	11.0	23.6	16.2	24.2	1

<sup>a</sup> Desired rotor preheat temperature set point.<sup>b</sup> Distillate.

Fatty acid profile of distillate (-d) and residue (-r).

Table 5

Main components-fatty acid profile-varying flow rate

Flow (g/min) <sup>a</sup>	20:1 Δ5-d	20:1 Δ5-r	22:1 Δ5-d	22:1 Δ5-r	22:1 Δ13-d	22:1 Δ13-r	22:2 Δ5,13-d	22:2 Δ5,13-r	Gardner Color <sup>b</sup>	Distillate to residue ratio
50	63.4	23.1	4.6	9.9	12.4	26.4	17.8	30.1	4	44.5
100	69.7	32.9	3.2	8.3	8.9	22.3	14.3	27.7	1	3.2
125	72.1	38.9	2.9	7.3	8	19.8	13.1	25.5	1	1.9
150	72.2	46.9	2.5	2.5	7.1	16.9	11.8	22.8	1	1.0

<sup>a</sup> Desired rotor preheat temperature set point.<sup>b</sup> Distillate.

Rotor temperature constant at 400 °C, rotor preheat 130 °C, and degas chamber 150 °C. Fatty acid profile of distillate (-d) and residue (-r).

Table 6

Meadowfoam distillation-ideal conditions

Distillation pressure (Pa)	Degasser pressure (Pa)	Rotor temperature (°C)	Rotor residue (°C)	Rotor preheat (°C)	Degasser chamber (°C)	Distillate to residue ratio	Degasser feed (°C)	Degasser drain (°C)	Distillate line (°C)	Residue line (°C)	Rotor feed pump (°C)	Flow (g/min)	Gardner Color <sup>a</sup>
<0.009	0.91	450	152–157	125	150	2–3	65	66	45	80	110	150	1

<sup>a</sup> Distillate.

Average set points for meadowfoam fatty acids distilled over 31 h. Ideal conditions for maximum throughput without lowering distillate quality.

the rotor and can range anywhere from room temperature to 800 °C (Fig. 2). Thus, the easiest place to make a significant impact on heat available for distillation is in the distillation chamber. A set of conditions where the rotor temperature was varied while other system conditions remained constant is shown in Table 1. The flow rate of the feed stock was set at 100 g/min. The rotor temperature was changed from 275 to 475 °C as the data points were collected. As the temperature increased, the distillate to residue ratio also increased, which was expected. At a rotor temperature of 475 °C, most of the material was distilled and only a fraction was collected as residue. With the increased temperature, color bodies also distilled. The Gardner color for the distillation products with varying rotor temperature are listed in Table 2. The 475 °C distillation had a Gardner color = 4, whereas the ideal Gardner color is 1. The increased rotor temperature, unfortunately, maximized the amount of distilled material at the sacrifice of color.

The fatty acid profile of both the residue and distillate are given in Table 2. The crude meadowfoam main components were 5-eicosenoic acid (59.9%), 5,13-docosadienoic acid (17.3%), 5-docosenoic acid (4.3%), and 13-docosenoic acid (11.9%). The distillation at a rotor temperature of 425 °C resulted in the closest percent composition to the crude meadowfoam fatty acids.

Different ratios of fatty acids were distilled depending on the rotor temperature. For the two rotor temperature extremes of 275 and 475 °C, the amount of 5-eicosenoic acid was enriched in the distillate to 72.0% at 275 °C from 59.2%, whereas the 5,13-docosadienoic acid was enriched in the distillate to 17.5% at the upper temperature of 475 °C from 10.1%.

With the effects of rotor temperature established, other variables were examined to help eliminate the cost of maintaining a high rotor temperature. The rotor preheat was varied from 100 to 150 °C while holding the rotor at 400 °C and other parameters constant as described previously. The upper limit for the rotor preheat was determined to be about 160 °C for a flow rate of about 110 g/min. The rotor preheat area was located between the rotor pump and the distilla-

tion chamber. The entire area was wrapped with heating tape and insulating tape. The temperature was taken at the point of entrance into the distillation chamber.

As the rotor preheat was increased from 100 to 150 °C, distillate-to-residue ratio also increased as it did with increased rotor temperature (Table 3). At a rotor preheat temperature of 150 °C, the ratio of distillate-to-residue was 6.9, as compared with 4.0 at 130 °C. The rotor preheat temperature varied dramatically based on the flow rate of the feed stock. As the flow changed, the rotor preheat changed also, an observation which led to using the rotor preheat as a way to monitor flow rate changes. The flow rate was measured periodically and adjusted with the metering pump and valve. By increasing the rotor preheat temperature, the distillate-to-residue ratio increased by a factor of 1.5 with a 20 °C change from 130 to 150 °C. An increase of 25 °C in the rotor temperature, 400–425 °C, gave an increase by a factor of four in the distillate-to-residue ratio (Table 1). When comparing the two possibilities, the rotor temperature seems to be the most efficient place to add energy into the system. Maintaining a steady rotor preheat temperature was difficult at the upper temperature limits when flow rates were high, so optimal temperature was determined to be about 125 °C.

The fatty acid profile varied as the rotor preheat changed (Table 4). As the temperature increased from 100 to 150 °C, the percentage of 5-eicosenoic acid decreased. By comparing just the two rotor preheat temperature extremes of 100 and 150 °C, the amount of 5-eicosenoic acid was enriched in the distillate to 65.5% at 100 °C from 59.5%, whereas the 5,13-docosadienoic acid was enriched in the distillate to 16.2% from 13.4% at the upper temperature of 150 °C. This was similar to the results obtained with an increase in rotor temperature, but not to the same extent. The higher the input temperatures the greater the amounts of C22 distilled. The percentage of 5-eicosenoic acid obtained between the temperature extremes, 100–150 °C, only varied by about 10%. There was no real effect on the Gardner color as the rotor preheat temperature changed and no real advantage to running the rotor preheat at the upper limits.



The degas chamber was the second most important component requiring heat on the distillation unit. Factors such as the size of the unit and the desired temperatures could result in large heat requirements for a particular distillation. By heating the degas chamber to high temperatures, the energy demands on the rotor and rotor preheat were lessened, but only at a thermal cost to the material. The material could remain in the chamber for minutes depending on the amount of material in the chamber and the exchange rate. Therefore, having a very high degas chamber temperature would negate the advantages of thin film distillation. However, heating the degas chamber correctly could ease the burden on the other heating systems, thereby improving distillation and saving energy spent on heating tapes. One of the advantages of the Myers system is the digital outputs, which display not only the set point, but also the actual temperature and the percent power that the heating device receives. Direct correlations exist between the temperature of the degas chamber and the percent power required to keep the rotor preheat at 130 °C (Fig. 3). As the degas chamber temperature increases,

the demand for rotor preheat energy lessens because passing the material through the degas chamber in a sense preheats it. Ideally the percent power should never stay at 100% for an extended time. Fig. 3 showed that the degas chamber temperature was optimized at about 155 °C so that the percent power on the rotor preheat ranged between 45 and 50% for a flow rate of about 100 g/min (Table 3). No general trend was observed for the fatty acid profile or split of the distillate and residue with changing degas temperature. Degas chamber temperature did not affect the temperature of the rotor preheat, thus no real differences in distillations were observed. The degas chamber was used to help eliminate the energy demands on the heating tapes and it aided in the degassing of the feed material.

The fastest and most effective way to alter meadowfoam distillation was to vary the flow rate (Table 5). By starving the distillation for material, the transfer tubes and pumps rapidly warmed to the desired set points. As the flow rate increased from 50 to 150 g/min, the distillate-to-residue ratio decreased, which was expected

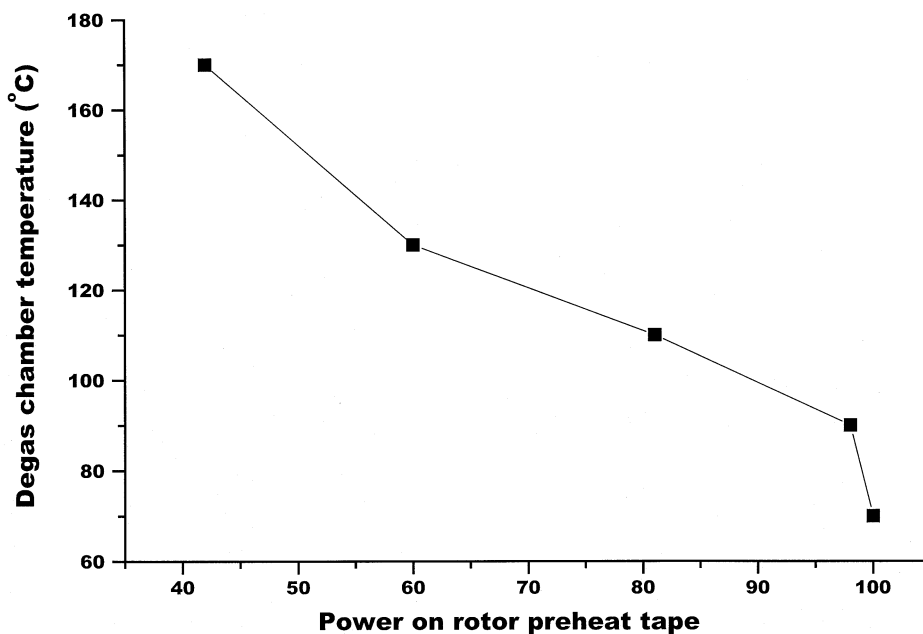


Fig. 3. Effects of degas chamber temperature on rotor preheat temperature to maintain 130 °C at a flow rate of 100 g/min.

because more of the material was passed through the system, more energy would be needed to maintain the ratio. By comparing just the two flow rate extremes of 50 and 150 g/min, the amount of 5-eicosenoic acid can be enriched in the distillate to 72.2% at 150 g/min from 63.4%, whereas the 5,13-docosadienoic acid can be enriched in the distillate to 17.8% at the upper flow rate, 50 g/min, from 11.8%. The distillate to residue split ratio varied greatly over the 50–150 g/min rate with the 100 g/min being the optimal distillation condition under these set of conditions. With the slower flow rate, the distillate to residue split was increased, but at the expense of the Gardner color. The 100 g/min flow rate demonstrated to have the best color, fatty acid profile, split ratio, and flow at a rotor temperature of 400 °C, rotor preheat 130 °C, and degas chamber 150 °C.

Being a pilot plant molecular unit, one of the main concerns is having a high throughput while maintaining the quality of the material. When the rotor temperature is increased to 450 °C, the flow can also be increased to 150 g/min while maintaining a distillate to residue split ratio between 2 and 3 (Table 6). The Gardner color and fatty acid profiles of the ideal conditions are the same as with the lower rotor temperature, 400 °C, and slower flow rate of 100 g/min.

#### 4. Conclusions

Meadowfoam fatty acids were effectively separated by a centrifugal molecular distillation unit. The precise distillation conditions were determined by varying conditions to obtain material that was light in color, Gardner color = 1. Conditions were then chosen to minimize high energy demands on any one element of the system. The varying conditions described were used to determine the ideal distillation conditions (Table 6). All of the conditions played a vital role in conducting a successful distillation. The rotor temperature and flow rate had the greatest impact on the

Gardner color and the fatty acid composition of the distillate. After the ideal conditions were determined, an additional larger volume (95 l) of meadowfoam fatty acids were distilled to verify the conditions.

#### Acknowledgements

Jason E. Adkins and Mark E. Klockenga provided expert technical assistance. The Fanning Corporation provided the crude meadowfoam fatty acids.

#### References

- Batistella, C.B., Maciel, M.R.W., 1996. Modeling, simulation and analysis of molecular distillators: centrifugal and falling film. *Computers Chem. Eng.* 20, S19–S24.
- Batistella, C.B., Maciel, M.R.W., 1998. Recovery of carotenoids from palm oil by molecular distillation. *Computers Chem. Eng.* 22, S53–S60.
- Biehler, R.M., Hickman, K.C.D., Perry, E.S., 1949. Small laboratory centrifugal molecular still. *Anal. Chem.* 21, 638–640.
- Firestone, D. (Ed.), 1994. *Color, Gardner 1963 (Glass Standards): Official and Tentative Methods of the American Oil Chemists Society*, fourth ed. American Oil Chemists' Society, Champaign, IL, Td 1a-64.
- Frykman, H.B., Isbell, T.A., 1999. Decolorization of meadowfoam estolides using sodium borohydride. *J. Am. Oil Chem. Soc.* 76, 765–767.
- Isbell, T.A., Plattner, B.A., 1997. A highly regioselective synthesis of  $\delta$ -lactones from meadowfoam fatty acids. *J. Am. Oil Chem. Soc.* 74, 153–158.
- Isbell, T.A., Kleiman, R., Plattner, B.A., 1994. Acid-catalyzed condensation of oleic acid into estolides and polyestolides. *J. Am. Oil Chem. Soc.* 71, 169–174.
- Isbell, T.A., Abbott, T.A., Carlson, K.D., 1999. Oxidative stability index of vegetable oils in binary mixtures with meadowfoam oil. *Ind. Crops Prod.* 9, 115–123.
- Isbell, T.A., Abbott, T.A., Dworak, J.A., 2000. Shampoos and Conditioners Containing Estolides. US Patent 6,051,214.
- Micov, M., Lutisan, J., Cvengros, J., 1997. Balance equations for molecular distillation. *Sep. Sci. Technol.* 32, 3051–3066.
- Rees, G.J., 1980. Centrifugal molecular distillation-II. *Chem. Eng. Sci.* 35, 841–845.